

Declaration

THE UNITED STATES PATENT AND TRADEMARK OFFICE

IN RE PCT NATIONAL STAGE APPLICATION OF  
WAGNER ET AL.

Group Art Unit: 1609

Examiner: HAVLIN, ROBERT H

Serial No.: 10/511,852

Filed: OCTOBER 14, 2004

For: AMINOPHENYLBENZOTHAZOLE  
COMPOUNDS

DECLARATION UNDER RULE 132

I, Barbara Wagner, a citizen of GERMANY, residing in Lörrach, hereby declare:

1. That I was awarded the degree of master (chemistry) from the University of Munich in 1991,
2. That I have been employed by Ciba Specialty Chemicals as a chemist since 1998,
3. That I presently hold the position of Head of Laboratory (Synthesis, Home & Personal Care),
4. That I consider myself an expert in the field of preparation of actives in cosmetic applications.
5. That I prepared the tested substances and tested the solubility herein described in strict accordance with my statements in the Declaration.

## Determination of the solubility of UV filters for specific solvents used for cosmetic formulations

### General

Determination of solubility in different solvents was carried out by stirring at 25°C.

If the viscosity of the solvents is too high the solubility had to be determined without a tempering bath at room temperature with a magnetic stirrer.

### Equipment:

- Beaker 10ml,
- tempering bath with magnetic stirrer
- centrifugal
- UV-VIS-Spectrometer
- Eppendorff pipette
- volumetric flask (V = 5ml-250ml)

### Accomplishment

2ml of the corresponding solvent were pipetted in a beaker.

Then 0.02g (1%) of the corresponding substance was weighted out and filled into the beaker together with the magnetic stirrer.

Then the suspension was placed in the tempering bath and the samples were stirred with

- water for 24h,
- ethanol or other volatile solvents for 6h and
- cosmetic solvents and oils for 7 days at 25°C.

When 1% of the substance is dissolved completely in the solvent, further 4% were added.

When these 5% are dissolved, substance was added until a saturated solution was obtained.

After the corresponding stirring time (6h, 24h, 7 days) the samples were filled in centrifugal glass and centrifuged for 20min at 10000UpM.

The overhang was filled into a small beaker with a pipette.

If the sample was still cloudy after centrifugation it was filtered through a Millex-LCR filter 0,5µm Non-Steril (Cat.No. SLCR 025 NZ).

The solubility was measured in a spectrometer or diluted with the corresponding solvent.

water: . with water

volatile solvents: with the corresponding solvent

cosmetic solvents: with dioxan

### **Calculation**

The concentration was determined from the extinction and the extinction coefficient determined before.

$$\text{formula: } c[\text{mol/l}] = \frac{E * f}{\epsilon * d}$$

E = extinction

f = dilution factor

$\epsilon$  = extinction coefficient

d = layer thickness of the cuvette.

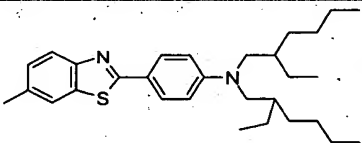
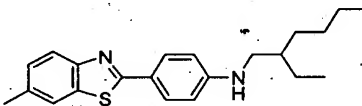
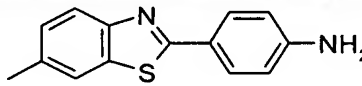
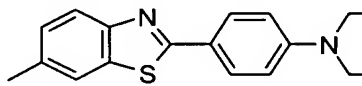
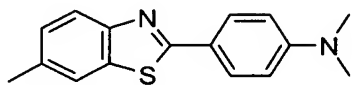
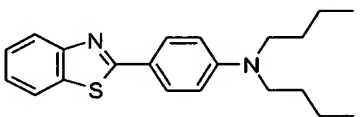
formula at solutions tel quel:  $c [\text{mol/l}] = E / \epsilon * d$

### **Solvents used for UV-Absorber Screening**

1. Finsolv TN
2. Miglyol 812N
3. Propylenglycol
4. Mineral Oil (Parafin, Merck)
5. Polydecene (Nexbase 2006 FG)
6. Cyclomethicone (DC345)
7. Water
8. Jojoba Oil

### **Results**

The results are listed in Table 1.

	<u>UV-Filter substance</u>		<u>Used solvents</u>						
	According to the present invention: A1, A2								
	Prior art: StdT 1, StdT 2, StdT 3, StdT 4								
	Structure		Water [%]	Cyclomethicone [%]	Finsolve TN [%]	Mygliol [%]	Mineral Oil [%]	Polydecene [%]	Jojoba Oil [%]
A1			0	15,9	50,4	50,6	50,1	50,6	51,1
A2			0	0,6	42,4	45,2	38,6	9,2	37,3
StdT 1			0,0	0,004	0,565	0,856	0,015	0,072	0,018
StdT 2			0,0	0,043	1,747	0,563	0,068	0,065	0,066
StdT 3			0,0	0,008	0,064	0,061	0,042	0,062	0,037
StdT 4			0,00	0,89	28,83	22,35	9,11	4,90	17,22

The tested solvents (except water) are predestinated for cosmetic sunscreen formulations.

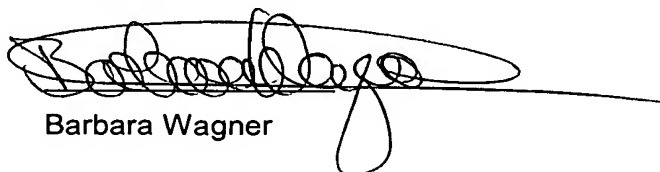
6. I, Barbara Wagner, further declare that it can be seen from the results listed in the Table above that the compounds A1 and A2 according to the present invention surprisingly show a solubility in cosmetic solvents, which is a multiple higher than the corresponding solubility values of the compounds of the closest prior art StdT 1, StdT 2, StdT 3 and StdT 4.

It is well known for a person of ordinary skill in the art that the sun protecting effect is dependent on the kind of formulation, i.e. the solvent used as well as the concentration of the UV filter in the formulation. Appropriate concentrations are in the range from 1 to 10 % b.w..

The test results show that the UV-Filter according to the present invention are very effective actives in sunscreen formulations because of their outstanding lipophilic properties expressed in solubility values > 1, compared to UV filters of the closest prior art which are useless because of their poor lipophilic properties.

7. I, Barbara Wagner, finally declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under section 101 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issuing thereon.

Signed the 20<sup>th</sup> day of September 2007

  
Barbara Wagner